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Indian Standard
SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(*Second Revision*)

Second Reprint FEBRUARY 2008
(Including Amendment Nos. 1, 2, 3, 4 & 5)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110 002

Indian Standard

SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(Second Revision)

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AMENDMENT NO. 1 NOVEMBER 1979
TO
IS:1824-1978 SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(Second Revision)

Alterations

(*Page 7, clause 3.1.1*) - Substitute the following for the existing clause:

'3.1.1 The packing shall also conform to the Tariff Rules as in force from time to time for packing of goods falling in the category of materials of low flash point.'

(*Page 25, clause E-2.1.1 to E-2.1.3*) - Substitute the following for the existing clauses:

'E-2.1.1 Weigh accurately a quantity of the material containing about 0.015 g of the lindane in a tared 250-ml erlenmeyer flask. Add 25 ml of isopropyl alcohol and shake the flask. Introduce 2.5 g of metallic sodium into the erlenmeyer flask, connect the flask to a reflux condenser and reflux for at least one hour on a water-bath. Shake the flask frequently during the refluxing period. Dissolve the excess metallic sodium by cautiously adding 10 ml of 50 percent isopropyl alcohol through the condenser at the rate of 1 to 2 drops per second. Boil for an additional 10 minutes and then add 60 ml of water.

E-2.1.2 Cool, add 2-3 drops of phenolphthalein indicator. Neutralize by adding dilute nitric acid dropwise and then add 10 ml in excess. Cool the flask to room temperature and quantitatively transfer the contents of the flask to a 250-ml separating funnel. Transfer the aqueous layer to a 500-ml erlenmeyer flask. Wash the contents of the separating funnel thrice using 20 ml water each time. Transfer the washings to the erlenmeyer flask. Reject the washed solvent. Mix well.

E-2.1.3 Add, with a pipette, 25 ml of standard silver nitrate solution. Coagulate the precipitated silver chloride on a water-bath for 30 minutes, cool the flask and, if necessary, filter the contents of the flask through a fast qualitative filter paper collecting the filtrate quantitatively in an erlenmeyer flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate as the case may be, and titrate the excess of silver nitrate with standard potassium thiocyanate solution (the end-point is the appearance of red ferric thiocyanate colour).'

(*Page 26, clause E-3.2, formula*) - Substitute the following for the existing formula:

Organic chlorine content of the material, percent by mass $= D = \frac{3.546 \times (B - A) \times N}{M} - C$

(AFCDC 6)

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AMENDMENT NO. 2 AUGUST 1982
TO
IS: 1824-1978 SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(Second Revision)

Addendum

(*Page 6, clause 2.2*) - Add the following new clause after 2.2:

"2.2.1 Pyrethrins, malathion and lindane employed in the formulation of insecticidal space spray (*see* Table 1), shall conform to IS:1051-1980 'Specification for pyrethrum extracts (*second revision*)', IS:1832-1978 'Specification for malathion, technical (*first revision*)', and IS:882-1956 'Specification for *Gamma*-BHC (Lindane)'."

(AFCDC 6)

Printed at Power Printers, New Delhi

AMENDMENT NO. 3 JANUARY 1986

TO

IS: 1824-1978 SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(Second Revision)

(*Page 6, clause 2.1*) - Substitute the following for the existing clause:

'2.1 Description and Identity

2.1.1 *Description* - The material shall be transprent and light coloured, kerosene based liquid with no unpleasant odour when sprayed.

2.1.2 *Identity* - If pyrethrin has been used in the formulation (*see* Table 1), then that formulation shall comply with Identity Test as described in Appendix A of IS:1051-1980* and shall not contain any synthetic pyrethroids. If any spots, in addition to those given by the standard reference sample are detected, it shall be ensured that these additional spots are not due to any synthetic pyrethroids. If the presence of any synthetic pyrethroids is established in the TLC testing, the total pyrethrin content as described in Appendix B of IS:1051-1980 should not be determined, as synthetic pyrethroids would analyse wrongly as pyrethrins.'

[*Page 6, clause 2.2* (*see also* Amendment No. 2)] - Add the following new clause below Table 1:

'2.2.2 *Tolerances on Active Ingredient* - When determined by the method prescribed in col 4 of Table 1, the observed individual active ingredient content, of any of the samples shall not differ by $^{+10}_{-5}$ percent (*m/m*) from the declared nominal value.

2.2.3 The actual value of the individual active ingredient in the formulation shall be calculated to one decimal more than the specified value in Table 1 and then rounded off to one decimal place less before applying the tolerances as stipulated in 2.2.1.

2.2.4 The average content of individual active ingredient, as applicable in all the samples taken shall not be less than the declared nominal value.'

(*Page 6, clause 2.3, line 2*) - Substitute 'IS:1448[P:20]-1982* *for* 'IS:1448[P:20]-1960*'

(*Page 6, clause 3.1*) - Substitute the following for the existing clause:

'3.1 Packing - The material shall be packed according to the requirements given in IS:8190(Part 3)-1979†.'

(*Page 6, foot-notes with '*' and '†' marks*) - Substitute the following for the existing foot-notes:

'*Methods of tests for petroleum and its products, P:20 Determination of flash point by Abel apparatus (*first revision*).

†Requirements for packing of pesticides:Part 3 Household pesticides.'

(*Page 7, clause 4.1 with Note*) - Substitute the following for the existing clause with Note:

'4.1 Representative samples of the material shall be drawn as prescribed in IS:10627-1983*.'

(*Page 7, foot-note*) - Add the following new foot-note:

'*Methods for sampling of pesticidal formulations.

(AFCDC 6)

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AMENDMENT NO. 4 NOVEMBER 1988

TO

IS : 1824 - 1978 SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(Second Revision)

[*Page* 6, *clause* **2.2.1** (*see also* Amendment No. 2)] — Substitute ' IS : 882-1984 ' *for* ' IS : 882-1956 '.

[*Page* 6, *clause* **2.2.1** (*see also* Amendment No. 3)] — Substitute ' **2.2.2** ' *for* ' **2.2.1** '.

[*Page* 6, *clause* **2.2.2** (*see also* Amendment No. 3)] — Substitute the following for the existing clause:

' **2.2.3** The actual value of the individual active ingredient in the formulation shall be calculated to two decimal places more than the specified value in Table 1 and then rounded off to one decimal place less, before applying tolerances as stipulated in **2.2.2**.'

[*Page* 6, *clause* **2.2.3** (*see also* Amendment No. 3)] — Substitute the following for the existing clause:

' **2.2.4** The average content of active ingredient as applicable in all samples taken shall not be less than the declared nominal value. '

(**AFCDC 6**)

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AMENDMENT NO. 5 JULY 1990
TO
IS 1824 : 1978 SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(*Second Revision*)

(*Page* 13, *clauses* **B-1.1** *to* **B-1.6**) — Substitute the following for the existing clauses:

'**B-1.1 Apparatus**

B-1.1.1 *Spectrophotometer or Photoelectric Colorimeter* — with a blue filter (420 nm) having 1-cm absorption cell.

B-1.2 Reagents

B-1.2.1 *Malathion Reference Standard* — of known purity.

B-1.2.2 *Kerosene* — distilled.

B-1.2.3 *Acetonitrile* — boiling range 80 to 82°C.

B-1.2.4 *Ethanol* — anhydrous.

B-1.2.5 *Sodium Hydroxide Solution* — 0·5 N.

B-1.2.6 *Ferric Reagent*

B-1.2.6.1 *Stock solution* — Dissolve 4·0 g of ferric chloride ($FeCl_3$, $6H_2O$) and 160 ml concentrated hydrochloric acid in water and dilute to 500 ml.

B-1.2.6.2 *Dilute solution* — Dilute 25 ml of the stock solution with water to 1 000 ml.

B-1.2.7 *Carbon Tetrachloride* — analytical reagent grade.

B-1.2.8 *Copper Reagent* — 1·5 percent (*m/v*). Prepare by dissolving 1·5 g of copper sulphate ($CuSO_4$, $5H_2O$) in 100 ml water.

B-1.3 Preparation of Standard and Sample Solutions

B-1.3.1 *Preparation of Standard Solution*

B-1.3.1.1 Weigh malathion reference standard of known purity and prepare accurately 1 percent (*m/v*) solution in kerosene. Take 5 ml of the solution with a pipette into a 100-ml volumetric flask, add 2·5 ml acetonitrile, shake for 5 seconds and make up the volume with ethanol.

B-1.4 Procedure

B-1.4.1 To obtain accurate results, it is important that the analysis of standard and sample solutions be carried out simultaneously through the following procedure without interruption.

B-1.4.2 Transfer a 5 ml aliquot each of malathion standard for analysis and sample for analysis into separate 250-ml separatory funnels. Add 2 ml sodium hydroxide solution and mix well by gentle swirling for 5-10 seconds. Allow the solution to stand for exactly 2 minutes. Immediately add 75 ml ferric reagent and mix well by swirling for 10 seconds. Allow the solution to stand exactly for 5 minutes. Add 50 ml carbon tetrachloride, allow the mixture to drain for 1 minute and add 2 ml copper reagent. Shake the mixture for exactly 1 minute.

B-1.4.3 Take clear solvent phase containing malathion standard and sample into two different absorption cells. Measure the absorbances at 420 nm using carbon teterachloride as blank.

B-1.5 Calculation

$$\text{Malathion content, percent by mass} = \frac{A}{B} \times P$$

where

A = absorbance of sample for analysis solution at 420 nm;

B = absorbance of malathion reference standard solution for analysis at 420 nm; and

$$P = \frac{\text{Mass, in g, of malathion reference standard taken for preparation of 1 percent (} m/v \text{) solution}}{\text{Mass, in g, of kerosene used in the preparation of 1 percent (} m/v \text{) solution of malathion reference standard}} \times \text{Percent purity of malathion reference standard'}$$

(FADC 1)

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Indian Standard

SPECIFICATION FOR INSECTICIDAL SPACE SPRAY

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 31 January 1978, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 The original standard published in 1961 was revised in 1971 to restrict the scope of the standard to some specific formulations in order to check them on chemical tests basis alone, as adoptability of biological tests for routine quality control was found not practically feasible. Now with the promulgation of Insecticides Act, 1968, the Registration Committee of Central Insecticides Board (CIB) has approved certain insecticides and their safe concentrations to be produced and marketed in the country as space sprays. The standard is therefore being again revised to bring its requirements in alignment with those stipulated by CIB.

0.3 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and tests for insecticidal space spray either in the form of single insecticide product or products based on insecticide mixtures with or without synergist, piperonyl butoxide.

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*Rules for rounding off numerical values (*revised*).

1.1.1 This standard does not cover the requirements of ready to use insecticides meant for spot application.

2. REQUIREMENTS

2.1 The material shall be transparent and light coloured, kerosene based liquid with no unpleasant odour when sprayed.

2.2 The material shall be according to any one of the compositions detailed in Table 1.

TABLE 1 COMPOSITION OF INSECTICIDAL SPACE SPRAY

(*Clauses* 2.2, 3.2 *and* 5.1)

Sl No	Constituents	Composition, Percent by Mass	Method of Test, Ref to Appendix
(1)	(2)	(3)	(4)
i)	Pyrethrins	0·2	A
ii)	Malathion	2·0	B
iii)	Allethrins	0·5	D
iv)	Lindane	0·1	E
v)	Pyrethrins	0·05	A and C
	Piperonyl butoxide	0·5	
vi)	Pyrethrins	0·05	A and E
	Lindane	0·05	
vii)	Pyrethrins	0·05	A and B
	Malathion	1·0	
viii)	Pyrethrins	0·02	A, C and E
	Piperonyl butoxide	0·5	
	Lindane	0·02	
ix)	Pyrethrins	0·02	A, B and C
	Piperonyl butoxide	0·5	
	Malathion	0·05	

2.3 Flash Point (Abel) — When determined by the method prescribed in IS : 1448 [P : 20]-1960* the flash point of the material shall be above 32°C.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirements given in IS : 8190 (Part III)†.

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*Methods of tests for petroleum and its products, P : 20 Flash point by Abel apparatus.

†Requirements for packing of pesticides: Part III Household pesticides (*under preparation*).

NOTE — Till such time, the standard under preparation is published, the matter shall be subject to agreement between the concerned parties.

3.1.1 The packing shall also conform to the Tariff Rules as in force from time to time for packing of goods falling in the category of materials of high flash point.

3.2 Marking — The container shall bear legibly and indelibly the following information in addition to the information required under the Insecticides Act and Rules:

a) Name of the material,

b) Name of the manufacturer,

c) Date of manufacture,

d) Batch number,

e) Net mass of contents,

f) Percentage composition of the material (m/m) (*see* Table 1), and

g) The minimum cautionary notice as worded in Insecticides Act and Rules.

3.2.1 The product may also be marked with Standard Mark

3.2.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard Methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time, the standard under preparation is published, the matter shall be as agreed between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods as referred to in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Items* (i), (v) *to* (ix) *and* B-2.1]

DETERMINATION OF TOTAL PYRETHRINS CONTENT IN THE INSECTICIDAL SPACE SPRAY EVEN IN COMBINATION WITH EITHER LINDANE OR PIPERONYL BUTOXIDE

A-1. APPARATUS

A-1.1 The assembly of the apparatus is shown in Fig. 1 and the various components are given in **A-1.1.1** to **A-1.1.8**.

A-1.1.1 *Steam Generator* (*A*) — a round bottom flask of one litre capacity, about two-thirds full of water and fitted with a two-hole cork carrying safety tube (*B*) and a steam outlet tube.

A-1.1.2 *Safety Tube* (*B*) — 760 mm long and open at both ends. The safety tube is so fitted in the rubber stopper that it nearly reaches the bottom of the steam generator (but does not touch it) when the stopper is inserted in the neck of the steam generator.

A-1.1.3 *Delivery Tube* (*C*)

A-1.1.4 *Asbestos Pad* (*D*) — having a 64-mm hole in the centre.

A-1.1.5 *Erlenmeyer Flask* (*E*) — 500 ml capacity.

A-1.1.6 *Distilling Head* (*F*) — constructed as shown in Fig. 2. The various dimensional requirements of the distilling head are also given in Fig. 2.

A-1.1.7 *Condenser* (*G*) — water cooled. The dimensional requirements of the condenser are given in Fig. 1.

A-1.1.8 *Separating Funnel* (*H*) — 500 ml capacity, pear-shaped.

A-2. REAGENTS

A-2.1 Ethanolic Sodium Hydroxide — approximately 0·5 N in ethyl alcohol.

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*Specification for water for general laboratory use (*second revision*).

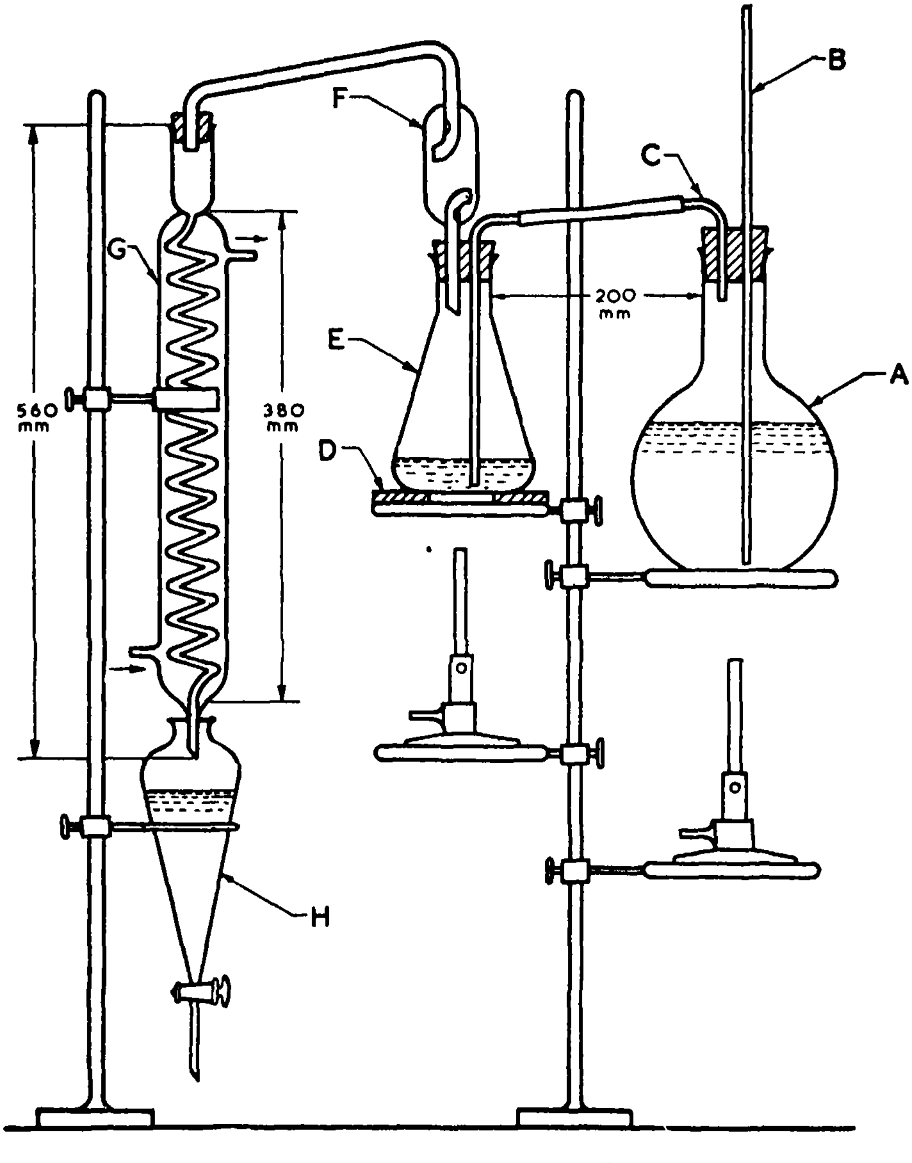
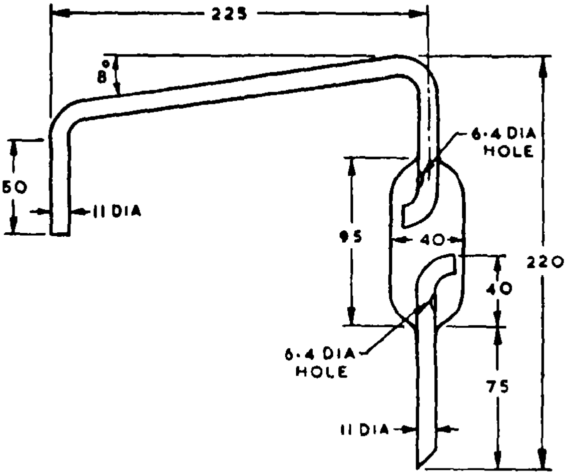


FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF TOTAL PYRETHRIN



All dimensions in millimetres.

FIG. 2 DISTILLING HEAD

A-2.2 Barium Chloride Solution — 10 percent (*m/v*).

A-2.3 Phenolphthalein Indicator Solution — one percent (*m/v*), in rectified spirit.

A-2.4 Dilute Sulphuric Acid — approximately 1 N.

A-2.5 Petroleum Ether — boiling point range 40 to 60°C, neutral to moist litmus paper.

A-2.6 Sodium Chloride — crystalline.

A-2.7 Standard Sodium Hydroxide Solution — approximately 0·02 N.

A-2.8 Concentrated Hydrochloric Acid — sp gr 1·16.

A-2.9 Ether

A-3. PROCEDURE

A-3.1 Weigh accurately a quantity of the material containing approximately 75 mg of total pyrethrins in a tared 500-ml Erlenmeyer flask. Add 20 ml ethanolic sodium hydroxide, or more if necessary, connect the flask to an upright, water cooled condenser and reflux the contents of the flask for at least one hour on a boiling water-bath. Transfer the contents of the flask, after cooling, to a 500-ml separating funnel. Remove the lower layer into a one-litre beaker, wash the funnel and its contents thrice with 15 ml of water every time, add the aqueous layer to the litre beaker, and discard the washed solvent. Dilute the solution contained in the beaker with water to about 200 ml. Add a few glass beads and reduce the volume of the solution to approximately 150 ml by boiling. Cool and transfer the solution quantitatively to 250-ml volumetric flask using water to wash the last traces of the solution into the flask. Add to the flask 10 ml of barium chloride solution, dilute to 250 ml mark with water and mix thoroughly. Filter the contents of the volumetric flask through a flutted filter paper. Reject the first portion of the filtrate if it is not clear Collect the clear filtrate in a 250-ml beaker. If it is necessary to stop at this stage, the filtrate should be left in alkaline condition only.

A-3.2 Determination of Pyrethrin I

A-3.2.1 Transfer exactly 200 ml of the clear filtrate (*see* **A-3.1**) into a 500-ml Erlenmeyer flask. Add one drop of phenolphthalein indicator solution to the contents of the flask, neutralize with dilute sulphuric acid and then add one millilitre in excess. Set the Erlenmeyer flask in position in the assembly of apparatus for the estimation of total pyrethrin (*see* Fig. 1). Distil with steam and collect the distillate in the separating funnel (*H*). Start heating the Erlenmeyer flask (*E*) with a Bunsen flame when the steam bubbles freely through the solution contained in it. Adjust the rate of distillation in such a way that 250 to 350 ml of the distillate will have collected in the separating funnel (*H*) when the volume of the solution in the Erlenmeyer flask (*E*) is reduced to approximately 20 ml. Stop the distillation at this stage. Immediately disconnect the delivery tube (*C*) from the steam generator (*A*). Reserve the residual solution contained in the Erlenmeyer flask (*E*) for the determination of Pyrethrin II (*see* **A-3.3**).

A-3.2.2 Add 50 ml of petroleum ether to the distillate contained in the separating funnel (*H*) and shake for a minute. If an emulsion is formed, add a few crystals of sodium chloride and shake for some time. Allow the layers to separate and draw off the aqueous layer into another 500 ml separating funnel containing another 50 ml of petroleum ether. Shake the contents of the second separating funnel for a minute and allow the layers to separate. Discard the aqueous layer. Wash the petroleum ether extract in the first separating funnel with 10 ml of water, transfer the separating wash-water to the second separating funnel and discard the wash-water after washing the petroleum ether extract contained in it. Repeat this process of washing with another portion of 10 ml of water. Combine the petroleum ether extracts in a single separating funnel and add to the combined extracts, 15 ml of water previously neutralized with standard sodium hydroxide solution using one drop of phenolphthalein indicator solution. Titrate the contents of the separating funnel with standard sodium hydroxide solution, shaking after each addition, until the aqueous layer just turns pink. Note the volume in millilitres of the standard sodium hydroxide solution used for the titration.

A-3.3 Determination of Pyrethrin II — Cool the Erlenmeyer flask (*E*) containing the residual solution from the steam distillation (*see* **A-3.2.1**) and filter the solution through a Gooch crucible. Wash the flask with three 10 ml portions of water and use the washings successively to wash the Gooch crucible. Transfer the filtrate and the washings quantitatively to a 500-ml pear shaped separating funnel. Add 5 ml of concentrated hydrochloric acid to the contents of the separating funnel and saturate with sodium chloride until it remains in excess. Extract the mixture by thoroughly shaking for one minute with 50 ml of ether, solvent. Allow the layers to separate. Draw off the aqueous layer into another separating funnel and extract with another 50 ml of ether, solvent. Continue this extraction and transfer the aqueous layer to a different separating funnel using 25 ml of ether, solvent, for the third and the fourth extractions. Successively wash the ether extracts contained in the different separating funnels with 10 ml of water. Repeat the washing using another 10 ml of water till free from acid. Combine the ether extracts in a single separating funnel and draw off any water that separated on standing. Filter the combined extracts through a plug of cotton wool previously wetted with the ether solvent. Wash the separating funnel and the plug of cotton wool with 10 ml of ether, solvent. Collect the filtrate and the washing in a 250-ml Erlenmeyer flask. Remove the ether from the Erlenmeyer flask either by distillation or by heating on a hot water-bath. If the ether is removed by heating on a hot water-bath, blowing a current of air into the Erlenmeyer flask twice during the heating, facilitates the removal of vapours. Place the Erlenmeyer flask in a water-bath maintained at 100 ± 1°C and dry the residue for 10 minutes. Add 30 ml of water, boil to dissolve the residue and allow to cool. Add a drop of phenolphthalein indicator solution to the contents of the Erlenmeyer flask and titrate with standard sodium hydroxide solution. Note the volume in millilitres of standard sodium hydroxide solution used for the titration.

A-4. CALCULATIONS

A-4.1 Calculate the total pyrethrin content of the material as follows:

a) Pyrethrin I content of the material, percent by mass $= X = \dfrac{41{\cdot}25\,AN}{M}$

b) Pyrethrin II content of the material, percent by mass $= Y = \dfrac{23{\cdot}38\,BN}{M}$

c) Total pyrethrin content of the material, percent by mass $= X + Y$

where

A = volume, in ml, of standard sodium hydroxide solution used for the titration of the extract containing Pyrethrin I (*see* **A-3.2.2**);

N = normality of standard sodium hydroxide solution;

M = mass, in g, of the material taken for the test (*see* **A-3.1**); and

B = volume, in ml, of standard sodium hydroxide solution used for the titration of the solution containing Pyrethrin II (*see* **A-3.3**).

APPENDIX B

[*Table* 1, *Items* (ii), (vii) *and* (ix) and *Appendix* A]

DETERMINATION OF MALATHION AND TOTAL PYRETHRINS CONTENT IN THE INSECTICIDAL SPACE SPRAY EVEN IN COMBINATION WITH PIPERONYL BUTOXIDE

B-0. GENERAL

B-0.1 In the household sprays containing malathion and pyrethrin, the method of test for determining pyrethrin II content in the formulation has to be modified as it is known by experience that malathion interferes in the determination of pyrethrin II content. A colorimetric method, therefore, is prescribed for the determination of pyrethrin II content while the method for the determination of pyrethrin I content is the same as prescribed in Appendix A.

B-1. DETERMINATION OF MALATHION CONTENT

B-1.1 Outline of the Procedure — The colorimetric method for the determination of malathion involves dissolution of malathion and the formulations in either ethanol or methanol as the preliminary step. In the above formulation, kerosene is used as the solvent. Both ethanol and methanol are immiscible with kerosene. Thus the solvent would interfere in the analysis of malathion. Therefore, as the preliminary step, malathion should be extracted with a suitable solvent which would in turn should be preferably soluble in either ethanol or methanol. Also, the solvent should be such that it extracts preferably only malathion leaving pyrethrins in the kerosene layer. Or, if the solvent also extracts pyrethrins, the extent to which pyrethrins are extracted, should be so negligible as to give the final sample for analysis practically colourless.

B-1.2 Apparatus

B-1.2.1 *Spectrophotometer or Photoelectric Colorimeter* — with a blue filter (420 nm) having 1-cm absorption cell.

B-1.3 Reagents

B-1.3.1 *Malathion* — pure.

B-1.3.2 *Acetonitrile* — boiling range 80 to 82°C.

B-1.3.3 *Ethyl Alcohol* — anhydrous.

B-1.3.4 *Sodium Hydroxide Solution* — 0·5 N. Dissolve 4·0 g of sodium hydroxide in 200 ml of water.

B-1.3.5 *Ferric Reagent*

B-1.3.5.1 *Stock solution* — Dissolve 4·0 g of ferric chloride ($FeCl_3.6H_2O$) and 160 ml of concentrated hydrochloric acid in water and dilute to 500 ml.

B-1.3.5.2 *Dilute solution* — Dilute 25 ml of the stock solution with water to 1 000 ml.

B-1.3.5.3 *Copper sulphate solution* — 1·5 percent (*m/v*). Prepare by dissolving 1·5 g of copper sulphate ($CuSO_4.5H_2O$) in 100 ml of water.

B-1.4 Preparation of Standard and Sample Solutions

B-1.4.1 *Preparation of Standard Solution*

B-1.4.1.1 Accurately weigh into a 5- or 10-ml beaker, 0·100 ± 0·020 g of pure malathion. Quantitatively transfer it to a 100-ml graduated flask with ethyl alcohol. Then dilute with ethyl alcohol, to approximately 5 ml below the volume mark. Allow the solution to come to a selected temperature. Record this temperature. Dilute the solution with ethyl alcohol to the mark and mix well. Call this as standard solution A.

NOTE — Standard solution A may be retained for daily use up to two weeks. It is very important, however, that it be adjusted to the temperature recorded above before aliquots are withdrawn.

B-1.4.1.2 Transfer a 15-ml aliquot of standard solution A to a 250-ml graduated flask. Add 2·5 ml acetonitrile and dilute with ethyl alcohol to approximately 5 ml below the volume mark. Retain the solution until sample solution (*see* **B-1.4.2**) is prepared and is ready for final dilution. At that time, dilute with ethyl alcohol to the mark and mix well. Call this as standard solution.

B-1.4.2 *Preparation of Sample Solutions*

B-1.4.2.1 *Extraction with acetonitrile* — Weigh accurately 100 ml of the sample and extract with 25 ml of acetonitrile in a separating funnel. Allow the layers to separate and run acetonitrile layer into a 250-ml volumetric flask. Extract the remaining material in the separating funnel twice using 25 ml of acetonitrile each time. Transfer acetonitrile extract into the volumetric flask. Dilute the contents of the volumetric flask with methanol up to the mark. Mix well. Transfer a suitable aliquot (which gives a malathion concentration of sample very near to the standard) of the diluted solution into a 250-ml volumetric flask and dilute the same to the mark with methanol. Treat this as sample for analysis.

NOTE — If more than one sample solution is being prepared for assay, then all sample solutions and the standard solution should be diluted to approximately 5 ml below the volume mark as prepared. All should be diluted to the volume mark at the same time.

B-1.5 Procedure

B-1.5.0 To obtain accurate results, it is important that the analysis of standard and sample solutions be carried out simultaneously through the following procedure without interruption.

B-1.5.1 Transfer a 25-ml aliquot of standard for analysis and one sample for analysis into separate 250-ml separating funnels. Add 2 ml of sodium hydroxide solution and mix well by swirling gently (do not shake) for 5 to 10 seconds. Allow the solution to stand for exactly 2 minutes.

B-1.5.2 Immediately add 75 ml of ferric reagent and mix well by swirling for 10 seconds. Allow the solution to stand for exactly 5 minutes. During this 5 minutes period, fill two 50-ml graduated flasks to the mark with carbon tetrachloride and after a lapse of 5 minutes, simultaneously transfer the carbon tetrachloride from each graduated flask to the respective separating funnels. Allow the flasks to drain for one minute.

NOTE — From this point onward, sample for analysis and standard for analysis should be handled separately. Carry each one through the next step without interruption.

B-1.5.3 Add 2 ml of copper reagent. Start shaking the separating funnel immediately after addition of copper reagent and shake it for exactly one minute. The malathion-copper complex is unstable in the aqueous phase. The copper reagent should be added with fast delivery pipette and the shaking should be started with practically no time lapse. Allow the phases to separate. As soon as separation occurs, discard the aqueous phase. Allow some solvent phase to rinse the stem of the separating funnel, then run the clear solvent phase into a small beaker for transferring to an absorption cell or run the clear solvent phase directly into the cell.

B-1.5.4 Measure the absorbance of the yellow solution within 5 minutes at 420 nm using carbon tetrachloride as the blank.

NOTE — Absorbance measurements of both standard and sample solutions should be made at the same fixed time after the end of the shaking period. The yellow colour of the copper-complex in carbon tetrachloride is unstable and hence the colour should be read within 5 minutes after extraction. During this time the absorbance reading drops very slowly.

B-1.6 Calculations

B-1.6.1 The absorbance measurements of the standard and sample solution should agree within ± 10 percent of each other.

B-1.6.2 Malathion content, percent by mass

$$= \frac{A}{B} \times \frac{m}{M} \times \frac{\text{dilution of standard} \times f}{\text{dilution of sample}}$$

where

A = absorbance of sample for analysis solution at 420 nm;

m = mass, in g, of pure malathion taken for the preparation of standard for analysis;

f = purity of the malathion used for the preparation of the standard solution;

B = absorbance of standard for analysis solution at 420 nm; and

M = mass, in g, of the material taken for the preparation of the sample for analysis.

B-2. DETERMINATION OF TOTAL PYRETHRINS

B-2.1 Pyrethrin I Content — Follow same procedure for determining percent pyrethrin I content as given in Appendix A and term the calculated value of pyrethrin I content as X.

B-2.2 Pyrethrin II Content

B-2.2.0 *Outline of the Procedure* — Pyrethrin II, on hydrolysis with sulphuric acid, yields methanol. The latter is distilled, oxidized, and the formaldehyde produced is determined colorimetrically after condensation with chromotropic acid. The method is rapid and has given reproducible results. It is presumed to be more specific than the other methods available at present for this purpose.

B-2.2.1 *Apparatus*

B-2.2.1.1 *Photoelectric colorimeter* — having 575 nm filter.

B-2.2.2 *Reagents*

B-2.2.2.1 *Petroleum ether*

B-2.2.2.2 *Standard sulphuric acid solution* — 18 N.

B-2.2.2.3 *Potassium permanganate reagent* — Dissolve 3 g in 15 ml of 85 percent phosphoric acid and dilute to 100 ml with distilled water.

B-2.2.2.4 *Sodium bisulphite*

B-2.2.2.5 *Chromotropic acid reagent* — Prepare daily a solution containing 0·1 g of sodium 4, 5-dihydroxy naphthelene-2, 7 disulphomate in 4 ml of distilled water and add 46 ml of 12 M sulphuric acid.

B-2.2.2.6 *Standard methanol solution* — Dilute 30 mg methanol to 100 ml with distilled water.

B-2.2.3 *Procedure*

B-2.2.3.1 Weigh accurately the amount of sample containing about 0·075 g total pyrethrins in the steam distillation flask. Add 20 ml standard sulphuric acid and steam distil till about 30 ml of aqueous distillate is collected in the separating funnel. Steam distil with this some kerosene (used as the solvent in the formulation) as well. Transfer aqueous distillate to a 50-ml volumetric flask. Wash the kerosene layer with 10 ml of distilled water and transfer to the volumetric flask.

B-2.2.3.2 Pipette 10 ml of standard methanol solution into another 50-ml volumetric flask. Add 30 ml distilled water to it.

B-2.2.3.3 Place both flasks in an ice-bath. Allow sufficient time so that the solutions may cool to about 2°C and add to each flask 1 ml of ice-cold permanganate reagent. Mix well the contents of the flask, keeping them immersed in the ice-bath. Two minutes after the addition of the permanganate reagent, destroy the excess of the oxidant by addition of 0·1 g sodium bisulphite to each solution. Take the two flasks out of the ice-bath, let their contents come to room temperature and adjust the volume of each to 50 ml with distilled water.

B-2.2.3.4 Prepare a reagent blank by treating 40 ml of distilled water with potassium permanganate and sodium bisulphite as described above.

B-2.2.3.5 Determine the formaldehyde content of each solution, by placing 1 ml in a 10-ml volumetric flask, and adding sufficient amount of the chromotropic acid reagent to bring the volumes of the solutions to 10 ml. Cover each flask with a glass-stopper, mix well and immerse in a boiling water-bath for 30 minutes. Cool the contents of each flask to room temperature, transfer to the cells of the colorimeter and measure the optical densities at 560 nm (or at 575 nm). Subtract the value of the blank from those of the sample and the standard.

B-2.2.3.6 Calculate the amount originally present in 1 ml of the sample used, and multiply with 50 to obtain the total amount of methanol produced during the hydrolysis of pyrethrin II. Relate this figure to the pyrethrin II content of the sample by assuming that 32 mg methanol are produced by 368 mg pyrethrin II.

B-2.2.3.7 The final result may be obtained directly from the corrected optical densities using the above formula.

B-2.2.4 *Calculations*

$$\text{Pyrethrin II content, percent by mass} = Y = \frac{A_{smp} \times m \times 0{\cdot}115}{A_{std} \times M}$$

where

A_{smp} = corrected optical density of the sample;

m = concentration of standard methanol solution as prepared and expressed as mg of methanol per 100 ml;

A_{std} = corrected optical density of the standard; and

M = mass, in g, of the sample taken for analysis.

Total pyrethrin content, percent by mass $= X + Y$

where

X = pyrethrin I content, and

Y = pyrethrin II content.

APPENDIX C

[*Table* 1, *Items* (v), (viii) *and* (ix)]

DETERMINATION OF PIPERONYL BUTOXIDE CONTENT IN THE INSECTICIDAL SPACE SPRAY

C-1. APPARATUS

C-1.1 Photoelectric Colorimeter or Spectrophotometer — capable of measuring absorbance at 625-635 nm.

C-2. REAGENTS

C-2.1 Tannic Acid — purified.

C-2.2 Piperonyl Butoxide — purified.

C-2.3 Acetic Acid

C-2.4 Phosphoric Acid

C-2.5 Tannic Acid Solution — prepared by dissolving 0·025 g tannic acid (**C-2.1**) in 20 ml acetic acid and making the volume to 100 ml with phosphoric acid. Shall be freshly prepared daily.

C-2.6 Piperonyl Butoxide Solution — prepared by dissolving 1·00 g of piperonyl butoxide (*see* **C-2.2**) in 100 ml volumetric flask with deodorized kerosene. Ten millilitres of this solution, when further diluted to 200 ml in deodorized kerosene will give a solution containing 50 µg of piperonyl butoxide solution/0·1 ml. If standard is to be used with sample containing pyrethrins or malathion, a quantity of pyrethrins or malathion to give ratio to sample of these ingredients shall be added to standard before initial dilutions.

C-3. PROCEDURE

C-3.1 Weigh accurately 0·1 ml of the sample and add 5 ml tannic acid solution (*see* **C-2.5**) in 18 × 150 mm test tube. Shake vigorously for 1 minute. Treat standard and blank, consisting of 0·1 ml deodorized kerosene, simultaneously in the same manner.

C-3.1.1 Place test tubes in test-tube basket and immerse in vigorously boiling water-bath for 5 minutes. Remove basket and let tubes cool to room temperature. Transfer solutions to colorimeter tubes and read, against water, using 625-635 nm filter or setting.

C-3.1.2 Subtract absorbance reading of deodorized kerosene from readings of both sample and standard.

C-4. CALCULATION

$$\text{Piperonyl butoxide content, percent by mass} = \frac{A_{smp} \times \text{purity of standard} \times 0{\cdot}5}{A_{std} \times 100}$$

where

A_{smp} = absorbance of sample, and

A_{std} = absorbance of standard.

APPENDIX D

[*Table* 1, *Item* (iii)]

DETERMINATION OF ALLETHRIN IN INSECTICIDAL SPACE SPRAY

D-0. PRINCIPLE

D-0.1 Allethrin reacts quantitatively with ethylenediamine to form chrysanthemum monocarboxylic acid which is determined by titration with standard sodium methylate in a pyridine medium. Chrysanthemum monocarboxylic acid, the anhydride, and this acid chloride interfere quantitatively and are determined independently.

D-1. REAGENTS

D-1.1 Ethanol — anhydrous.

D-1.2 Phenolphthalein Indicator — 1 percent in ethanol.

D-1.3 Standard Sodium Hydroxide — 0·02 N and 0·1 N.

D-1.4 Methanol — anhydrous.

D-1.5 Methanolic Hydrochloric Acid Standard Solution — 0·1 N. Dilute 17 ml of 6 N hydrochloric acid to 1 000 ml with methanol in a volumetric flask. Standardize against 0·1 N sodium hydroxide (*see* **D-1.3**) using phenolphthalein as the indicator (*see* **D-1.2**).

D-1.6 Morphine Solution — Dilute 8·7 ml of redistilled morphine to 1 000 ml with methanol in a reagent bottle. Fit the bottle with a two-holed rubber stopper and through one hole insert a 20-ml pipe so that the tip extends below the surface of the liquid; through the other hole insert a short piece of glass tubing to which is attached an aspirator bulb.

D-1.7 Ethylenediamine, Redistilled — Water content 3 percent maximum.

D-1.8 Dimethyl Yellow-Methylene Blue Mixed Indicator — Dissolve 1·0 g of dimethyl yellow (*p*-dimethylaminoazobenzene) and 0·1 g of methylene blue in 125 ml of methanol.

D-1.9 Alpha-naphtholbenzein Indicator — 1·0 percent in alcohol.

D-1.10 Thymolphthalein Indicator — 1·0 percent in pyridine solution.

D-1.11 Sodium Methylate Standard Solution — Transfer 25 ml of 4 N sodium methylate to a one-litre reagent bottle containing 75 ml of methanol and dilute to one litre with redistilled pyridine. Standardize this solution against benzoic acid using pyridine as a solvent and thymolphthalein as indicator. The reagent readily absorbs carbon dioxide from the air, and is best preserved and used in a 50-ml automatic burette. All vents open to the air must have protective Ascarite tubes.

D-2. PROCEDURE

D-2.1 Determination of Chrysanthemum Monocarboxylic Acid Chloride

D-2.1.1 Add 8 to 10 drops of the dimethyl yellow-methylene blue indicator to approximately 150 ml of methanol in a suitable container, and add 0·1 N methanolic hydrochloric acid dropwise, until the solution appears reddish brown by transmitted light.

D-2.1.2 Add 0·02 N methanolic potassium hydroxide dropwise until the appearance of the first green colour.

D-2.1.3 Transfer 25 ml of this solution to each of three 125-ml glass-stoppered Erlenmeyer flasks.

D-2.1.4 Reserve one of the flasks as a reference colour for the end-point determination.

D-2.1.5 Into each of the other flasks introduce 1·5 to 2·5 g of sample weighed to the nearest 0·1 mg using a suitable weighing pipette. Swirl the flask during the addition of the sample.

D-2.1.6 Within 5 minutes, titrate each of the samples with 0·02 N methanolic potassium hydroxide to the first green end-point, using the blank as a reference colour.

D-2.1.7 *Milliequivalent Calculation*

$$C = \frac{A \times N}{M}$$

where

C = chrysanthemum monocarboxylic acid chloride, milliequivalent per g;

A = ml of KOH required for sample titration;

N = normality of KOH; and

M = mass in g of the sample taken for test.

D-2.1.8 *Percentage Calculation*

Chrysanthemum monocarboxylic acid chloride, percent by mass $= C \times 18{\cdot}67$

D-2.2 Determination of Chrysanthemum Monocarboxylic Acid

D-2.2.1 Transfer 25 ml of the anhydrous ethanol to each of two 125-ml glass-stoppered Erlenmeyer flasks.

D-2.2.2 Add 8 or 9 drops of alpha-naphtholbenzein indicator to each flask and cool the flask and contents to 0°C in a suitable ice-bath.

D-2.2.3 Neutralize by dropwise addition of 0·02 N sodium hydroxide to a bright green end-point.

D-2.2.4 Into each flask introduce 1·5 to 2·5 g of sample weighed to the nearest 0·1 mg using a suitable weighing pipette.

D-2.2.5 Immediately titrate with standard 0·02 N sodium hydroxide to the first bright green end-point.

D-2.2.6 *Milliequivalent Calculation*

$$D = \frac{A \times N}{M}$$

where

D = acid and acid chloride, milliequivalent per g;
A = ml of NaOH required (*see* **D-2.2.5**);
N = normality of NaOH; and
M = mass, in g, of sample taken for the test.

D-2.2.7 *Percentage Calculation*

$(D - C) \times 16{\cdot}82$ = chrysanthemum monocarboxylic acid, percent by mass; and
C = chrysanthemum monocarboxylic acid, milliequivalent per g (*see* **D-2.1.7**).

D-2.3 Determination of Chrysanthemum Monocarboxylic Anhydride

D-2.3.1 Carefully pipette 20 ml of the morphline solution into each of four 250-ml Erlenmeyer flask, using the same pipette for each transfer. Fill the pipette by exerting pressure in the reagent bottle with the aspirator bulb.

D-2.3.2 Reserve two of the flasks for blanks.

D-2.3.3 Into each of the other flasks introduce 1·5 to 2·5 g of sample weighed to the nearest 0·1 mg using a suitable weighing pipette.

D-2.3.4 Swirl the flask and allow the samples to stand together with the blanks at room temperature for 5 minutes.

D-2.3.5 Add 4 or 5 drops of the dimethyl yellow-methylene blue indicator to each of the flasks and titrate with standard 0·1 N alcoholic hydrochloric acid until the colour changes from green to a faint red when viewed by transmitted light.

D-2.3.6 *Milliequivalent Calculation*

$$E = \frac{(B - A) \times N}{M}$$

where

E = chrysanthemum monocarboxylic anhydride, milliequivalent per g;

B = ml of HCl required for the blank;

A = ml of HCl required for the sample;

N = normality of HCl used; and

M = mass, in g, of sample taken for the test.

D-2.3.7 *Percentage Calculation*

$(E - 2C) \times 31{\cdot}84$ = chrysanthemum monocarboxylic anhydride, percent by mass

where

C = chrysanthemum monocarboxylic acid chloride, milliequivalent per g (*see* **D-2.1.7**).

D-2.4 Determination of Allethrin

D-2.4.1 Introduce an amount of sample calculated to contain 0·8 to 1·1 g of allethrin into each of two 250-ml Erlenmeyer flasks. Weigh the sample to the nearest 0·1 mg using a suitable weighing pipette.

D-2.4.2 Reserve two additional flasks as blanks.

D-2.4.3 To each of the blanks and samples introduce 25 ml of the redistilled ethylenediamine using a suitable dispensing burette protected from the atmosphere by ascarite tubes. Swirl the samples to effect solution.

D-2.4.4 Allow the samples to stand together with the blanks at 25 ± 2°C for 2 hours.

NOTE — The reaction may also be conducted in heat-resistant pressure bottles at 98 ± 2°C for 30 minutes.

D-2.4.5 Wash down the sides of each flask with a 50-ml portion of redistilled pyridine.

D-2.4.6 To each flask add 6 to 10 drops of the thymolphthalein indicator and titrate with standard 0·1 N sodium methylate in pyridine to the first permanent blue-green end-point. (With colourless samples, the first blue end-point may be used.)

D-2.4.7 *Milliequivalent Calculation*

$$F = \frac{(A - B) \times N}{M}$$

where

F = allethrin, milliequivalent per g;
A = ml of sodium methylate required for the sample;
B = ml of sodium methylate required for the blank;
N = normality of sodium methylate; and
M = mass, in g, of sample taken for the test.

D-2.4.8 *Percentage Calculation*

Allethrin, percent by mass = $(F + C - D - E) \times 30.24$

where

C = chrysanthemum monocarboxylic acid chloride, milliequivalent per g (*see* **D-2.1.7**);
D = acid and acid chloride, milliequivalent per g (*see* **D-2.2.6**); and
E = chrysanthemum monocarboxylic anhydride, milliequivalent per g (*see* **D-2.3.6**);

APPENDIX E

[*Table* 1, *Items* (iv), (vi) *and* (viii)]

DETERMINATION OF LINDANE IN INSECTICIDE SPACE SPRAY

E-1. REAGENTS

E-1.1 Isopropyl Alcohol — of two concentrations, one 99 percent and the other 50 percent by volume.

E-1.2 Metallic Sodium — pure, in the form of ribbon or cut in small pieces.

E-1.3 Phenolphthalein Indicator Solution — one percent (*m/v*) in rectified spirit.

E-1.4 Dilute Nitric Acid — 50 percent by volume.

E-1.5 Standard Silver Nitrate Solution — approximately 0·1 N.

E-1.6 Ferric Ammonium Sulphate Solution — saturated, aqueous, freshly prepared.

E-1.7 Standard Potassium Thiocyanate Solution — approximately 0·1 N.

E-1.8 Concentrated Nitric Acid – sp gr 1·42.

E-2. PROCEDURE

E-2.1 Determination of Total Chlorine Content

E-2.1.1 Weigh accurately a quantity of the material containing about 0·15 g of the lindane in a tared 250-ml Erlenmeyer flask. Add 25 ml of isopropyl alcohol and shake the flask. Introduce 2·5 g of metallic sodium into the Erlenmeyer flask, connect the flask to a reflux condenser and reflux for at least one hour on a water-bath. Shake the flask frequently during the refluxing period. Dissolve the excess metallic sodium by cautiously adding 10 ml of 50 percent isopropyl alcohol through the condenser at the rate of 1 to 2 drops per second. Boil for an additional 10 minutes and then add 60 ml of water.

E-2.1.2 Cool, add 2-3 drops of phenolphthalein indicator. Neutralize by adding dilute nitric acid dropwise and then add 10 ml in excess. Cool the flask to room temperature and quantitatively transfer the contents of the flask to a 250-ml separating funnel. Transfer the aqueous layer to a 500-ml volumetric flask Wash the contents of the separating funnel thrice using 20 ml water each time. Transfer the washings to the volumetric flask. Reject the washed solvent. Dilute the solution in the volumetric flask to the mark with water. Mix well.

E-2.1.3 Pipette a 50-ml aliquot of the diluted solution in 250-ml conical flask. Add, with a pipette, 25 ml of standard silver nitrate solution. Coagulate the precipitated silver chloride on a water-bath for 30 minutes, cool the flask and, if necessary, filter the contents of the flask through a fast qualitative filter paper collecting the filtrate quantitatively in a conical flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate as the case may be, and titrate the excess of silver nitrate with standard potassium thiocyanate solution (the end-point is the appearance of red ferric thiocyanate colour).

E-2.1.4 Carry out a blank determination on the reagents using the above methods.

E-2.2 Determination of Inorganic Chlorine

E-2.2.1 *Qualitative Test for Chlorides* — Shake about 25 ml of the material with 10 ml water in a separating funnel. Separate the layers and to the aqueous layer add a few drops of concentrated nitric acid and 1 ml of approximately 2 percent (*m/v*) silver nitrate solution. If a white turbidity or opalescence appears, indicating the presence of inorganic chlorine, quantitatively determine the inorganic chlorine in the material.

E-2.2.2 *Quantitative Determination of Inorganic Chlorine* — Weigh accurately, by difference, about 10 g of the material in a separating funnel. Extract twice with water using 15 ml each time. Transfer the aqueous extracts to a 250-ml conical flask. Add 5 ml of concentrated nitric acid to the combined aqueous extracts and add 25 ml of standard silver nitrate solution. Coagulate the precipitate as before and titrate the excess silver nitrate with standard potassium thiocyanate solution.

E-2.2.3 Carry out a blank determination on the reagents as before.

E-3. CALCULATIONS

E-3.1 Inorganic chlorine content, percent by mass

$$= C = \frac{35\cdot46 \times (B - A) \times N}{M}$$

where

B = volume, in ml, of standard KCNS (potassium thiocyanate) solution required for the blank;

A = volume, in ml, of standard KCNS solution required for the sample;

N = normality of standard KCNS solution; and

M = mass, in g, of the sample taken for the test.

E-3.2 Organic chlorine content of the material, percent by mass

$$= D = \frac{35\cdot46 \times (B - A) \times N}{M} - C$$

where

B = volume, in ml, of standard KCNS solution required for the blank during the determination of total chlorine content;

A = volume, in ml, of standard KCNS solution required for the sample during the determination of total chlorine content;

N = normality of the standard KCNS solution;

M = mass, in g, of the sample taken for analysis during the determination of total chlorine content; and

C = inorganic chlorine content, percent by mass (*see* **E-3.1**).

E-3.2.1 Lindane content of the material, percent by mass = 1 368 × total organic chlorine content (D) (*see* **E-3.2**)

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